Variable Temperature Studies of Zeolite Zn-A

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Abstract No. read3626
Beamline(s): **X3B1**

Introduction: Understanding the behavior of zeolites during the dehydration process is important as this is nearly always undertaken before zeolites can be used for applications such as catalysis, separations or as micro-reactors for the production of new nanomaterials. The dehydration process can be simple, involving the loss of water that is acting either in a pore-filling role or as loosely coordinated to the intrapore cations, or complex involving hydrolysis and formation of hydroxide and/or oxide ions. Zinc-exchanged zeolite A is a system that clearly displays complex behavior, as we have recently shown from the analysis of high resolution synchrotron X-ray data from a sample dehydrated at 500 ºC [1]. More recently, we have been studying the behavior of Zn-A using in-house *in situ* powder X-ray diffraction. We observe structural changes between *ca.* 150-200 ºC in air or under helium, consistent with a lowering of the symmetry from cubic to rhombohedral. This is followed by a further change at higher temperatures, *ca.* 600 ºC, back to cubic symmetry. In order to better understand this process, we have undertaken higher resolution *in situ* studies at beamline X3B1.

Methods and Materials: The sample of Zn-exchanged zeolite A was produced using standard ion exchange procedures. A sample was loaded into a thin walled 0.7 mm diameter glass capillary which was sealed at one end and left open to the atmosphere by inserting a plug of glass wool into the other. The capillary was inserted into the furnace inside the hutch at beamline X3B1 and diffraction data collected at temperature at a wavelength of 0.7004 Å using a crystal analyzer configuration.

Results: Data from the region of 12–14º (2θ) at selected temperatures are shown in Figure 1. Least-squares fitting of the peak profiles, Table 1, shows a progressive broadening of the peaks that reaches a maximum value at 180 ºC, and a continuous decrease in intensity of the (800) reflection at *ca.* 13.25º. At this temperature, there are also clearly two peaks in the region of 13.7º, where the (644) and (820) reflections overlap. This behavior is consistent with that observed previously, although it is not yet clear what structural changes are occurring. Further analysis is in progress. With continued heating, the (800) reflection disappears and there is a notable decrease in peak widths, suggesting a return to cubic symmetry. This is not expected to occur until *ca.* 600 ºC based on previous experiments, and the reason for the drastic difference in temperature is not yet clear.

Acknowledgments: We would like to thank Peter Stephens for experimental assistance, the Royal Society for the award of a University Research Fellowship (PAA) and EPSRC for the provision of a studentship (JER).

References: [1] J.E. Readman, I. Gameson, J.A. Hriljac, P.P. Edwards and P.A. Anderson, "Synthesis and structure of zinc oxide clusters encapsulated in zeolite LTA," Chemical Communications, 595, 2000.

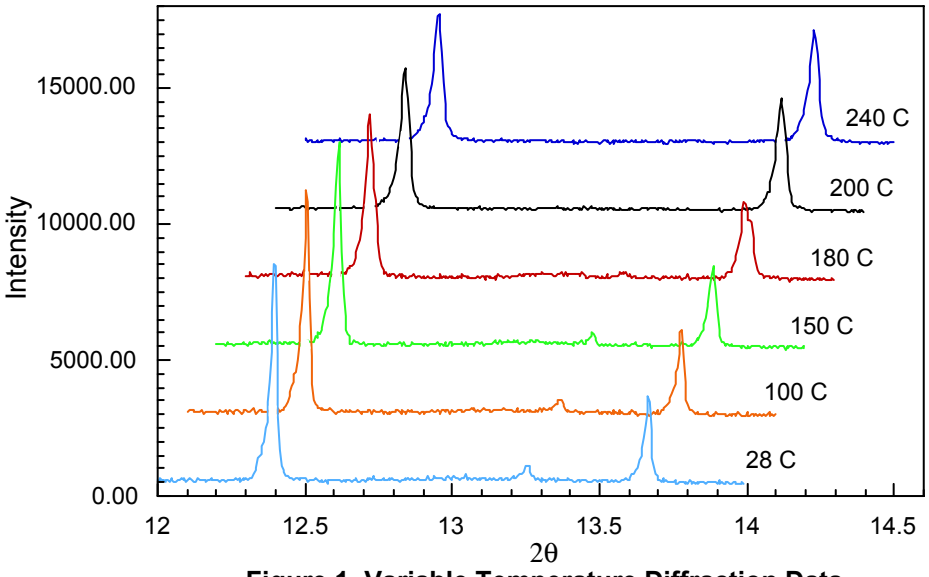

Figure 1. Variable Temperature Diffraction Data.

Table 1. Fitted Peak Widths.

Temp (ºC)	*FWHM (º)*
28	0.0152
100	0.0163
150	0.0208
180	0.0359
200	0.0300
240	0.0272